Center for By-Products Utilization

DESIGN AND TESTING CONTROLLED LOW-STRENGTH MATERIALS (CLSM) USING CLEAN COAL ASH

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CBU Report No. CBU-1997-07
REP-287
December 1996

For publication in the ASTM STP 1331 "The Design and Application of Controlled Low-Strength Materials (Flowable Fill)" 1997.

Also for presentation in the ASTM Symposium on "The Design and Application of CLSM (Controlled Low Strength Materials)" June 1997.

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REFERENCE: Naik[1], T. R., Kraus[2], R. N., Sturzl[3], R. F., and Ramme[4], B. W., "Design and Testing Controlled Low-Strength Materials(CLSM) Using Clean Coal Ash," The Design and Application of Controlled Low-Strength Materials (Flowable Fill), ASTM STP 1331, A. K. Howard and J. L. Hitch, Eds., American Society for Testing and Materials, 1997.

ABSTRACT: The major objective of this project was to develop mixture proportions for controlled low-strength material (CLSM) using clean coal ash obtained from atmospheric fluidized bed combustion (AFBC). A clean coal ash is defined as the ash derived from SO_X and NO_X control technologies. The specific ashes used for this project were: (1) circulating fluidized bed boiler fly ash and bottom ash and (2) stoker-type boiler fly ash and bottom ash. These two coal ash samples were characterized for physical and chemical properties. Chemical properties and water leaching tests were also performed on the hardened CLSM. Many initial CLSM mixtures were developed by blending the two types of ash.

Tests conducted on the final three selected CLSM mixtures included compressive strength, bleeding, setting and hardening, settlement, length change of hardened CLSM, permeability, mineralogy, and chemical water leach testing. Results show that acceptable CLSM material can be developed by blending the fluidized bed boiler ash with the stoker boiler ash. Recommendations for a pilot scale manufacturing application of the three CLSM mixtures were made based upon the lab test results.

KEYWORDS: controlled low-strength material (CLSM), mixture proportion, compressive strength, permeability, shrinkage, leachates, fluidized bed boiler, coal ash

The project objective was the development of controlled low-strength material (CLSM), namely manufactured dirt, and mixtures using clean coal ash obtained from AFBC boilers. A clean coal ash is defined as ash derived from SO_X and NO_X control technologies. Two types of ash material were used for this project: Ash A, a stoker-type boiler fly ash and bottom ash and Ash B, circulating fluidized bed boiler fly ash and bottom ash. This paper summarizes results of the laboratory research activities for the project. No cement was used for CLSM mixtures using Ash B because of the cementitious nature of the fluidized bed boiler ash, and to make better use of these by-products.

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CLEAN COAL AND CONVENTIONAL ASH MATERIAL

Two different types of ash were used in this project: (1) Ash A, a conventional fly ash and bottom ash obtained from stoker type boilers and (2) Ash B, a clean coal ash obtained from fluidized bed boilers.

Ash A was obtained from the combustion residues of three stoker-type boilers. A Hazard/Elkhorn blend of coal was used for these units. Fly ash and bottom ash from these boiler units were combined and stored in a silo.

Ash B consisted of ash produced from a 20-MW circulating fluidized bed boiler. This fluidized bed boiler burns a coal blend consisting of 20% Southern Illinois bituminous coal and 80% of petroleum coke. Ash B is classified as a clean coal ash. Calcium carbonate in the form of limestone or dolomite (Basu and Fraser 1991, Podolski 1984, Tung and Williams 1987, and Yerushalmi 1986) was injected into the fluidized bed boiler during combustion of the coal/coke combination to act as a sorbent for SO_2 emissions. The limestone breaks down to an oxide form while heated (Podolski 1984):

$$CaCO_3 \rightarrow CaO + CO_2$$

The calcium oxide then reacts with the SO_2 which is produced during coal combustion to form calcium sulfate:

$$CaO + SO_2 + 1/2\ O_2 \rightarrow CaSO_4$$

The advantages of the fluidized bed boiler are a significant reduction (85 to 98%)(Tung and Williams 1987, and Yerushalmi 1986) of SO_2 emissions without the utilization of a wet scrubber system typically used in flue gas desulfurization (Podolski 1984), and reduced NO_x emissions since coal combustion temperatures are lower than those typically found in a pulverized coal boiler (Tung and Williams 1987, and Yerushalmi 1986). Ash B material consisted of the spent limestone material and bottom ash along with fly ash from the fluidized bed boiler unit.

Characterization of Ash Materials

Physical test results on each source of ash are shown in Table 1. The tests results given in Table 1 are those typically required by ASTM Specification for Coal Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete (C 618). Table 2 shows the results of the sieve analysis of the ashes. Ash A material had larger size fractions (approximately 6% retained No. 4 sieve) and was more uniformly graded than Ash B. Seventy percent of Ash B material was between No. 50 and No. 200 sieve. Both Ash A and Ash B contain approximately 20% material finer than No. 200 sieve.

Elemental Analysis of Ash A and Ash B

The results of the elemental analysis of Ash A and Ash B samples are given in Table 3. These ashes were analyzed using instrumental neutron activation analysis. The neutron activation analysis method exposes the sample to neutrons which results in an activation of many elements. This activation consists of radiation of various elements. For the ashes used for this project, gamma ray emissions were detected.

Many different elements may be detected simultaneously based on the gamma ray energies and half-lives.

TABLE 1--Physical test results.

TEST	Ash A	Ash B	ASTM C 618 SPECIFICATIONS	
			Class C	Class F
Retained on No.325 sieve, (%)	66.5	54.8	34 Max	34 Max
Strength Activity Index with Cement at 7 days, (% of Control)	54.4	49.2	75 Min	75 Min
Strength Activity Index with Cement at 28 days, (% of Control)	57.5	53.5	75 Min	75 Min
Water Requirement (% of Control)	130	103	105 Max	105 Max
Specific Gravity	2.00	2.58	-	-
Variation from Mean, (%)				
Fineness (R325)	3.5	1.3	5 Max	5 Max
Specific Gravity	2.0	2.7	5 Max	5 Max

TABLE 2--Sieve analysis of ashes.

Sieve Size	Percent Passing		ASTM C 33 Specifications for Fine Aggregates
	Ash A	Ash B	
12.5 mm	97.8	-	-
9.5 mm	97.0	-	100
4.75 mm (No. 4)	93.6	99.4	95-100
2.36 mm (No. 8)	89.6	99.2	80-100
1.18 mm (No. 16)	83.7	98.6	50-85
600 ìm (No. 30)	65.9	95.1	25-60
300 ìm (No. 50)	46.3	91.9	10-30
150 ìm (No. 100)	33.9	63.9	2-10
75 ìm (No. 200)	21.1	21.9	-

TABLE 3--Elemental analysis of ashes.

ELEMENTAL (BULK CHEMICAL) ANALYSIS (mg/kg unless noted otherwise)		
Element	Ash Type	
	Ash A	Ash B
Aluminum (Al)	90472.3	11708.3
Antimony (Sb)	7.9	2.9
Arsenic (As)	306.0	113.7
Barium (Ba)	187.4	61.7
Beryllium (Be)	3.7	19.0
Boron (B)	79.0	78.0
Cadmium (Cd)	<1520.5*	<585.6*
Calcium (Ca)	594.6	40581.5
Chloride (Cl)	65.5	1781.0
Chromium, Total (Cr)	75.5	26.7
Cobalt (Co)	46.4	7.0
Copper (Cu)	233.1	282.1
Fluoride (F)	2.5	13.0
Iron (Fe)	22610.0	13525.0
Lead (Pb)	33.0	18.0
Magnesium (Mg)	6101.8	2098.2
Manganese (Mn)	607.8	1561.8
Mercury (Hg)	27.3	4.0
Molybdenum (Mo)	<66.3*	125.7

* Less than detection limit noted.

TABLE 3 (Cont.)--Elemental analysis of ashes.

ELEMENTAL (BULK CHEMICAL) ANALYSIS (mg/kg unless noted otherwise)		
Element	Ash Type	
	Ash A	Ash B
Nickel (Ni)	2097.0	54055.0
Nitrogen, Soluble $NO_2 + NO_3$	1.8	1.7
Phosphorous (P)	340.0	200.0
Potassium (K)	14385.0	2894.5
Selenium (Se)	673.0	223.3
Silicon (Si)	3900.0	9300.0
Silver (Ag)	18.5	3.3
Sodium (Na)	1166.5	4552.5
Strontium (Sr)	123.3	<11.2*
Thallium (Tl)	1.3	<0.3*
Tin (Sn)	260.6	103.4
Vanadium (V)	200.8	3086.8
Zinc (Zn)	12.9	10.1

* Less than detection limit noted.

The elemental composition of the two types of ash differed considerably. Ash B contained higher quantities of beryllium, calcium, chloride, flouride, manganese, molybdenum, nickel, silicon, sodium, and vanadium and lower amounts of aluminum, arsenic, barium, cadmium, chromium, cobalt, iron, lead, magnesium, mercury, potassium, selenium, strontium, and silver. They contained comparable amounts of antimony, boron, copper, nitrogen, phosphorus, thallium, tin, and zinc.

The elemental analysis of ash is dependent on the type of coal used, combustion process, additives, and so forth. Although general elemental composition is of interest, the elements that may leach are of greater relevance. Leachate results of the hardened CLSM are discussed later in this paper.

Ash A and Ash B - Oxides, Sulfite, Loss of Ignition, and Mineralology

A summary of the oxides, sulfite, and loss on ignition (LOI) analysis is given in Table 4. Ash A had a very high LOI, approximately 25% by weight. Ash B also had a high (15%) LOI. Although the high LOI ashes would not be suitable for use in structural concrete, the high LOI should not affect its application to CLSM since air-entraining admixtures are generally not required to be used for manufacturing CLSM.

TABLE 4––Analysis for oxides, sulfite, and loss on ignition.

Analysis Parameter	Ash A, %	Ash B, %	ASTM C 618 Requirements	
			Class C	Class F
Silicon Dioxide, SiO_2	41.1	6.3	--	--
Aluminum Oxide, Al_2O_3	23.5	3.0	--	--
Iron Oxide, Fe_2O_3	3.4	1.7	--	--
$SiO_2 + Al_2O_3 + Fe_2O_3$	68.1	11.1	50.0 Min	70 Min
Calcium Oxide, CaO	1.2	37.2	--	--
Magnesium Oxide, MgO	0.6	0.8	--	--
Titanium Oxide, TiO_2	1.3	0.0	--	--
Potassium Oxide, K_2O	1.3	0.2	--	--
Sodium Oxide, Na_2O	0.3	0.6	--	--
Sulfite, SO_3	0.7	32.3	5.0 Max	5.0 Max
Loss on Ignition, LOI	24.8	15.0	6.0 Max	6.0 Max
Moisture, %	0.6	0.4	3.0 Max	3.0 Max

Water requirement for the ash with high LOI generally is higher, but again, this should not impact its use for CLSM. The very high value of LOI of these ashes may be due to incomplete combustion or low temperature combustion characteristics of a typical low-mix boiler. A visual examination of Ash A material revealed that there were pieces of incompletely burned coal which were partially covered with slag. This is not unusual for the bottom ash material of stoker boilers.

Mineralology of the Ash A and Ash B are shown in Table 5. The mineralogical composition of the two ashes are different. This is to be expected since two different types of boilers (fluidized bed and stoker) produced these ashes. Ash B also contained a significant amount (42%) of anhydrite $CaSO_4$. This is not a mineral typically found from pulverized coal ash boilers. Ash B anhydrite $CaSO_4$ is due to the use of limestone as a sorbent for controlling SO_X emissions in the fluidized bed boiler. Ash B also contained calcite, 13.2%; portlandite, 1.5 %; and quartz, 2.1%. The free lime (CaO) content was only 2.3%. Oxide analysis (Table 4) indicates a total lime (CaO) of 37%. The total lime content indicates the total calcium (Ca^+) ions and oxide (O^-) ions present. The combination of Ca^+ and O^- can occur in minerals other than free lime such as anhydrite, calcite, and portlandite. Free lime indicates the quantity of lime in a compound form alone, without other elements. Some of these minerals may not be sufficiently cementitious.

Ash A contained quartz, 4.5%; mullite, 24.1%; and the remaining amounts as amorphous (glass phases) materials of 71.5%. The mineralogical composition of Ash A indicates that this ash may not have sufficiently independent cementitious properties.

TABLE 5––Mineralogy of ashes.

MINERALOLOGY, % by Weight		
Analysis Parameter	Ash A	Ash B
Quartz, SiO_2	4.5	2.1
Mullite, Al_2SiO_5	24.1	*
Gypsum, $CaSO_4 \cdot 2H_2O$	*	*
Anhydrite, $CaSO_4$	*	42.1
Bassanite, $CaSO_4 \cdot O \cdot 5H_2O$	*	*
Ettringite, $CaAl_2\ (SO_4, SiO_4, CO_3)\ (OH)12 \cdot 26H_2O$	*	*
Calcite, $CaCO_3$	*	13.2
Portlandite, $Ca(OH)_2$	*	1.5
Lime, CaO	*	2.3
Amorphous	71.5	38.7

* Not detected.

CLSM MIXTURES

Although CLSM may have strengths as high as 8.3 MPa at the 28-day age, a CLSM mixture that is considered to be excavatable at a later age using hand tools should have a compressive strength lower than 0.7 MPa at the 28-day age (American Concrete Institute 1994, American Coal Ash Association 1995, Canadian Portland Cement Association 1990, Kraus and Naik 1996, Naik and Ramme 1990, 1994, Naik et al. 1991, and Ramme et al. 1995). Mechanical equipment such as a backhoe is required for excavation at a later age for a mixture with compressive strengths higher than 0.7 MPa up to as high as 1.4 MPa. A mixture with compressive strength higher than 1.4 MPa may require jackhammers for excavation at a later age or may be chipped loose with a backhoe bucket.

Based upon the test results of a preliminary test phase (Kraus and Naik 1996), three mixtures were selected for further development and testing. They are:

(1) 100% Ash B, Mix 1 and 2,
(2) 50% Ash B + 50% Ash A, Mix 3 and 4, and
(3) 25% Ash B + 75% Ash A, Mix 5 and 6.

An exothermic reaction takes place when water is added to ash produced by the fluidized bed boiler. Free calcium oxide and calcium sulfite/sulfate produce the exothermic reaction (Basu and Fraser 1991):

$$CaO + H_2O \rightarrow Ca(OH)_2 + 15.6\ kCal/g\ mol$$
$$CaSO_4 + 2\ H_2O \rightarrow CaSO_4 \bullet 2H_2O + 4.22\ kCal/g\ mol$$

This exothermic reaction was particularly evident in the 100% Ash B mixtures.

Bench scale mixtures of approximately 0.13 m^3 were mixed for each of these test mixtures. Mixture proportions and rheological properties for these six mixtures are given in Table 6. The reported mixture proportions were adjusted based upon the actual yield of the test batch. Density of the CLSM decreased as the amount of Ash A was increased in the mixture. Water required was approximately the same for all mixtures. Flow/spread was measured in accordance with ASTM Provisional Test Method for Flow Consistency of Controlled Low Strength Material (PS 28). The water-to-cementitious materials ratio for bench scale mixtures was 0.56 ± 0.08.

Compressive Strength of CLSM Mixtures

Compressive strength test results for the CLSM are shown in Table 7. Three 150- by 300-mm cylinders were tested in accordance with ASTM Test Method for Preparation and Testing of Soil-Cement Slurry Test Cylinders (D 4832) at each test age. The 28-day compressive strength test results range from 0.7 to 4.5 MPa. The mixtures containing 75% Ash A (Mixes 5 and 6) had the lowest compressive strength of 0.7 MPa, and the 100% Ash B mixture (Mixes 1 and 2) had the highest values at 3.3 and 4.5 MPa. A range of values for compressive strength at the 28-day age of the CLSM mixture should be one of the considerations when specifying it for a given application if excavation is required. The CLSM mixture with the 28-day compressive strength of 0.7 MPa is considered to be easily excavated at the later day age. Excavation of a mixture of 4.5 MPa would involve a significantly higher cost.

Setting and Hardening of CLSM Mixtures

Setting and hardening characteristics measurements of the CLSM mixtures are reported in Table 8. The values reported are the average of three measurements taken from three 150- by 300-mm cylinders for each mixture. To evaluate their setting and hardening characteristics, the test specimens were left in the molds and not covered for the entire 14-day measurement period.

Setting characteristics of the CLSM mixtures were measured by applying a 4.5-kg force on a 50-mm-long seven penny nail and then measuring the depth of the nail penetration. The CLSM mixtures generally hardened slowly as the quantity of Ash A increased. These nail penetration measurements were taken from the test cylinders where the bleedwater was not removed periodically. Therefore, these setting times are considered a worst case for the CLSM mixtures for field use because the bleedwater will generally drain off to the surrounding soil from a CLSM mixture placed at a project site.

Bleedwater measurements were negligible for the mixture containing 100% Ash B, Mix 2. The maximum amount of bleedwater was for the mixtures with 75% Ash A + 25% Ash B, Mixtures 5 and 6, which had a 20-mm depth of bleedwater on the top of the 150-mm-high cylinder mold at the age of 1 h.

Settlement values, reported in Table 8, are the measurement to the "solids" portion of the CLSM mixture with respect to a datum (that is, top of the cylinder mold) taken as the original height of the cylinder. These settlement values are not indications of how the material may behave when a load is applied. Positive settlement (or an increase in the settlement) measurements of the CLSM indicate that the CLSM material actually expanded when hardening. The CLSM mixtures containing 100% Ash B (Mixes 1 and 2) exhibited expansion.

TABLE 6--Mixture proportions and fresh CLSM test results for bench scale mixtures.

Mixture No.	1	2	3	4	5	6
Ash A, Content (%)	0	0	50	50	75	75
Ash B, Content (%)	100	100	50	50	25	25
Ash A (kg/m^3)	0	0	485	485	670	665
Ash B (kg/m^3)	1150	1177	485	485	220	220
Cement (kg/m^3)	0	0	0	0	0	0
Water (kg/m^3)	555	570	550	550	560	555
Water-to-Cementitious Materials Ratio	0.48	0.48	0.57	0.57	0.63	0.63
Flow/Spread (mm)	230	355	230	280	245	255
Slurry Temp. (°C)	32	36	31	30	26	29
Air Temp. (°C)	22	24	19	24	24	24
Air Content (%)	1.6	1.5	3.0	3.4	3.9	4.9
Slurry Density (kg/m^3)	1710	1750	1520	1530	1450	1430
Test Mixture Yield(m^3)	0.13	0.05	0.13	0.05	0.13	0.13

TABLE 7--Compressive strength test results - bench scale CLSM mixtures.

Mixture No.	1	2	3	4	5	6
Ash A Content (%)	0	0	50	50	75	75
Ash B Content (%)	100	100	50	50	25	25
Cement Content (%)	0	0	0	0	0	0
Test Age (days)	Compressive Strength (MPa)					
3	1.7	-	1.1*	-	-	-
7	2.1	1.2	1.7	0.3	0.3	0.2
14	2.6	-	-	-	0.6	0.3
28	3.3	4.5	2.2	2.6	-	0.7

* Test at five day age.

TABLE 8––Setting and hardening characteristics - CLSM mixtures.

Ash Mixture	Test Age	50-mm Nail penetration, mm	Bleedwater, mm	Settlement, mm*
Mix 2 (100% Ash B)	1-Hour	50	0	0
	1-Day	5	0	+3
	3-Day	2	0	+5
	5-Day	0	0	+5
	7-Day	0	0	+3
	14-Day	0	0	+3
Mix 4 (50% Ash A + 50% Ash B)	1-Hour	50	16	-16
	1-Day	50	8	-16
	3-Day	24	3	-0.5
	5-Day	10	0	-14
	7-Day	5	0	-13
	14-Day	2	0	-13
Mix 5 & 6 (75% Ash A + 25% Ash B)	1-Hour	50	20	-20
	1-Day	50	14	-13
	3-Day	24	10	-16
	5-Day	10	6	-16
	7-Day	5	3	-14
	14-Day	3	0	-14

* Settlement values of the CLSM are taken from a datum level at the original cast height of the CLSM

Permeability of Hardened CLSM Mixtures

Permeability of the CLSM mixtures was measured at the age of approximately 30 days. Results are reported in Table 9. Three 100- by 100-mm cylinders were cast from each mixture for permeability measurements. At the age of seven days, the test specimens were immersed in 23°± 2°C lime-saturated water until the time of testing. A falling head parameter was utilized for these tests per ASTM Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter (D 5084). Typically, permeability of the CLSM increased as the amount of Ash A increased in the mixture, 1.3×10^{-6} cm/s for the 100% Ash B (Mix 1), to 3.1×10^{-6} cm/s for the 75% Ash A + 25% Ash B (Mix 6). These values are consistent with 100×10^{-6} to 10×10^{-6} cm/s for a compacted granular backfill and 0.1×10^{-6} cm/s or less for compacted clays. Differences in permeability between Mixes 3 and 4 can be attributed to the small quantity of CLSM utilized for the test (0.0008 m^3). Permeability of the mixtures is not expected to increase at later ages.

TABLE 9--Permeability of CLSM mixtures.

Mixture No.	Ash A Content,%	Ash B Content, %	Specimen No.	Test Age, days	Permeability, x 10^{-6} cm/s	
					Actual	Average
1	0	100	1 2 3	31 31 31	1.0 2.1 0.9	1.3
3	50	50	1 2 3	40 - 40	3.4 - 3.7	3.5
4	50	50	1	28	2.1	2.1
6	75	25	1 2 3	35 35 35	3.2 3.1 3.0	3.1

Oxides, SO_3 and Loss on Ignition of Hardened CLSM Mixtures

The hardened CLSM material was also analyzed for oxides, sulfite, loss on ignition, and moisture content. The results of the analysis, Table 10, show that the composition of the hardened CLSM material varies with the ash composition of the CLSM. A comparison with the oxide analysis of the ash material (Table 4) shows that the approximate oxide composition of the hardened CLSM material could be obtained by appropriately blending the two ash types. These test data are logical because the CLSM is produced by mixing the Ash A and Ash B materials with water without any other ingredients.

Mineralology of the Hardened CLSM

Mineralogy of the hardened CLSM material is presented in Table 11. Mineral formation of the CLSM mixtures are very different compared to the minerals of the ash used. The CLSM using 100% Ash B (Mix 1) is primarily composed of gypsum (~60%), amorphous glass phases (36%), and ettringite (5%). The CLSM mixture of 25% Ash B and 75% Ash A (Mix 5) has a much different composition with nearly 70% of the material amorphous, about 15% mullite, and 10% bassanite. With a high percentage of the 100% Ash B CLSM (Mix 1) being gypsum (Table 11), there may be potential for the Ash B material for uses other than CLSM where gypsum is desired.

Leachate Results of Hardened CLSM

The leachate results of the three mixtures tested are given in Table 12. The three mixtures are: 100% Ash B, Mix 1; 50% Ash A + 50% Ash B, Mix 3; and 25% Ash B + 75% Ash A, Mix 5. The leachate parameters shown are the elements which the Wisconsin Department of Natural Resources have identified in their interim guidelines for waste reuse in CLSM. Leachate results for pH, sulfate, total alkalinity, and total dissolved solids were relatively high, but the results are consistent with leachate from other circulating fluidized bed boiler ashes (Basu and Fraser 1991).

TABLE 10––Hardened CLSM mixtures - analysis for oxides, sulfite, and loss on ignition.

Analysis Parameter	CLSM Mix 1, 100% Ash B, %	CLSM Mix 3, 50% Ash B + 50% Ash A, %	CLSM Mix 5, 25% Ash B + 75% Ash A, %
Silicon Dioxide, SiO_2	8.5	17.1	26.0
Aluminum Oxide, Al_2O_3	4.5	10.0	15.5
Iron Oxide, Fe_2O_3	1.8	2.4	2.8
Calcium Oxide, CaO	30.4	19.7	12.0
Magnesium Oxide, MgO	0.7	0.7	0.6
Titanium Oxide, TiO_2	0.1	0.4	0.7
Potassium Oxide, K_2O	0.2	0.5	0.8
Sodium Oxide, Na_2O	0.4	0.5	0.5
Sulfite, SO_3	25.3	17.7	11.4
Loss on Ignition, LOI	25.6	27.9	30.4
Moisture, %	28.5	1.5	31.6

TABLE 11––Mineralogy of CLSM mixtures.

MINERALOLOGY, % by Weight			
Analysis Parameter	CLSM Mix 1, 100% Ash B	CLSM, Mix 3, 50% Ash B + 50% Ash A	CLSM Mix 5, 25% Ash B + 75% Ash A
Quartz, SiO_2	0.5	1.7	2.9
Mullite, Al_2SiO_5	--	6.5	14.7
Gypsum, $CaSO_4 \cdot 2H_2O$	58.9	16.9	--
Anhydrite, $CaSO_4$	--	6.5	0.5
Bassanite, $CaSO_4 \cdot O \cdot 5H_2O$	--	6.2	9.4
Ettringite, $CaAl_2$ (SO_4 ,SiO_4 ,CO_3) (OH)12$\cdot 26H_2O$	5.0	--	--
Calcite, $CaCO_3$	--	7.8	5.3
Portlandite, $Ca(OH)_2$	--	1.6	--
Lime, CaO	--	--	--
Amorphous	35.5	52.7	67.2

TABLE 12--Leachate analysis of hardened CLSM mixtures.

LEACHATE RESULTS, mg/l unless noted otherwise			
	CLSM Ash Mixture		
Leachate Parameter	Mix 1, 100% Ash B	Mix 3 50% Ash A + 50% Ash B	Mix 5 25% Ash B + 75% Ash A
Total Alkalinity	2300	1600	66
Aluminum (Al)	0.024	0.026	0.110
Antimony (Sb)	<0.004*	<0.004*	<0.004*
Arsenic (As)	<0.005*	<0.005*	<0.005*
Barium (Ba)	0.190	0.130	0.110
Beryllium (Be)	<0.001*	<0.001*	<0.001*
Boron (B)	<0.004*	<0.004*	<0.004*
Cadmium (Cd)	<0.0001*	<0.0001*	<0.0001*
Calcium (Ca)	1600	1100	640
Chloride (Cl)	34	37	20
Chromium, Total (Cr)	0.0021	0.0016	0.0027
Cobalt (Co)	<0.008*	<0.008*	0.008
Conductivity	11000 umhos/cm	7900 umhos/cm	2600 umhos/cm
Copper (Cu)	<0.002*	<0.002*	<0.002*
Fluoride (F)	0.92	0.61	<0.25*
Total Hardness	4100	2700	1600
Iron (Fe)	<0.006*	<0.006*	<0.006*
Lead (Pb)	<0.005*	0.006	<0.005*
Magnesium (Mg)	<0.02*	<0.02*	0.32
Manganese (Mn)	<0.002*	<0.002*	<0.002*
Mercury (Hg)	<0.0002*	<0.0002*	<0.0002*

* Less than detection limit.

TABLE 12(Cont.)--Leachate analysis of hardened CLSM.

LEACHATE RESULTS, mg/l unless noted otherwise			
	CLSM Ash Mixture		
Leachate Parameter	Mix 1, 100% Ash B	Mix 3, 50% Ash A + 50% Ash B	Mix 5 25% Ash B + 75% Ash A
Molybdenum (Mo)	0.650	0.610	0.380
Nickel (Ni)	<0.008*	<0.008*	<0.008*
Nitrite & Nitrate	1.9	4.5	0.5
pH	12.60 S.U.	10.36 S.U.	10.74 S.U.
Phosphorous (P)	<0.1*	<0.1*	<0.1*
Potassium (K)	16	35	40
Selenium (Se)	<0.0008*	<0.0008*	<0.0008*
Silicon (Si)	0.16	0.30	9.4
Silver (Ag)	<0.0004*	<0.0004*	<0.0004*
Sodium (Na)	36	49	33
Strontium (Sr)	0.910	1.500	2.100
Sulfate (SO_4)	1600	1300	1600
Thallium (Tl)	<0.001*	<0.001*	<0.001*
Tin (Sn)	<0.40*	<0.040*	<0.040*
Total Dissolved Solids	4200	3400	2600
TOC	2	2	2
TOX	0.052	0.180	0.089
Vanadium (V)	0.076	0.110	2.9
Zinc (Zn)	0.0073	0.012	0.0038

* Less than detection limit.

SUMMARY OF MIXTURE TEST RESULTS

The following is a summary of these results.

Three bench scale mixtures were selected for detailed testing based upon preliminary tests (Kraus and Naik 1996) for this project. None of the mixtures used cement. The three mixtures developed are:

(1) 100% Ash B, Mix 1,
(2) 50% Ash B and 50% Ash A, Mix 3, and
(3) 75% Ash A and 25% Ash B, Mix 5 or 6.

Density of the fresh CLSM increased as the amount of Ash B increased.

Compressive strength of the CLSM mixtures range from 0.7 MPa for the 25% Ash B + 75% Ash A mixture to 4.5 MPa for the 100% Ash B mixture. If the CLSM is expected to be excavated in the future, a lower strength CLSM at the 28-day should be used to minimize cost of excavating a mix with a compressive strength above 0.7 MPa at the 28-day age. Ash B, when mixed with water, was found to be expansive. This expansion was readily apparent for the 100% Ash B CLSM mixture. The expansive properties of the CLSM may be desirable in certain applications, for example, filling abandoned tunnels or tanks in which assurance of complete and tight fill would be desirable.

Permeability of the CLSM was low, approximately 10 to 100 times lower than that of compacted sand. The length change values after 7-day age show some expansion but not to the extent of the material at ages at less than 7 day (as measured by settlement values of the cylinders in their molds).

Oxide analysis of the hardened CLSM indicates that the oxide composition of the material is approximately the same as what would be obtained by blending the ashes. Minerals present in the CLSM materials are different than the original minerals depending upon the ash type as a result of amorphous glass materials in the ashes. The CLSM containing 100% Ash B contained nearly 60% gypsum, while nearly 70% of the material for the 25% Ash B + 75% Ash A mixture was amorphous (glass phase).

PROPOSED USAGE OF CLSM

Mixtures developed for this project could have distinctly different applications because of the variation in compressive strength. Also, as a result of the expansive nature of the 100% Ash B mixture, applications of this material should be judiciously evaluated to take into account for this characteristic. Expansion of the CLSM would be desirable for applications in which complete fill must be ensured such as tanks or abandoned tunnels. The 100% Ash B mixture should only be used where excavation would not be expected (because of high compressive strength of the mixture of 4.5 MPa at 28-day age).

If the potential for leaching is desired to be minimized, applications of the CLSM used in this study may be capped with concrete or asphalt or the CLSM may be used in a confined area.

Typical usage of this CLSM material will be for roadway sub-bases; backfilling of utility trenches; excavation for walls and buildings; filling abandoned sewer and tunnels; support for foundations; in addition to others.

REFERENCES

ACI 229R-94 Report, July 1994, "Controlled Low-Strength Materials (CLSM)," Concrete International, pp.55-64

American Coal Ash Association, Dec. 1995, Fly Ash Facts for Highway Engineers, Alexandria, VA

Basu, P., and Fraser, S. A., 1991, Circulating Fluidized Bed Boilers - Design and Operations, Butterworth-Heinemann, Stoneham, MA

Canadian Portland Cement Association, 1990, "Unshrinkable Fill for Utility Trenches in Streets," Concrete Information CPOO4.02P

Kraus, R. N. and Naik, T. R., Feb. 1996, "Development of Controlled Low Strength Materials ("Manufactured Dirt") Utilizing Manitowoc Public Utilities Ash," CBU Report to Manitowoc Public Utilities, Milwaukee, WI

Naik, T. R., and Ramme, B. W., Oct. 1990, "Low Strength Concrete and Controlled Low Strength Material (CLSM) Produced with High-Lime Fly Ash," in Proceedings of the CANMET/EPRI International Conference on Fly Ash in Concrete, Published by CANMET, Ottawa, Ontario

Naik, T. R., Ramme, B. W., and Kolbeck, H. J., Sep. 1991,"Controlled Low-Strength Material (CLSM) Produced with High-Lime Fly Ash," in Proceedings of the International Conference on the Utilization of Fly Ash and Other Coal Combustion By-Products, Shanghai Building Science Research Institute, Shanghai, China

Naik, T. R., and Ramme, B. W., 1994, "Low Strength Concrete and Controlled Low Strength Material (CLSM) Produced with Class F Fly Ash," in Controlled Low-Strength Materials, ACI SP-150, W. S. Adaska, Ed., American Concrete Institute

Podolski, W. F., 1984, "Fluidized Bed Combustion," in The Science and Technology of Coal and Coal Utilization, B. R. Cooper, and W. A. Ellingson, Eds., Plenum Press, New York

Ramme, B. W., Naik, T. R., and Kolbeck, H. J., June 1995, "Construction Experience with CLSM Fly Ash Slurry for Underground Facilities", in Proceedings of the Fifth CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete, Milwaukee, WI

Tung, S. E., and Williams, G. C., Jan. 1987, Atmospheric Fluidized Bed Combustion - A Technical Source Book, The Massachusetts Institute of Technology, Cambridge, MA

Yerushalmi, J., 1986, "An Overview of Commercial Circulating Fluidized Bed Boilers," in Circulating Fluidized Bed Technology, P. Basu, Ed., Pergamon Press, Ontario, Canada